#### TRANSLATION:



# FEDERAL REPUBLIC OF GERMANY

# **Priority Certification upon Filing of a Patent Application**

File No.:

P 44 05 983.3

Filing Date of Application: February 24, 1994

Applicant/Holder:

Henkel KGaA, Düsseldorf/DE

Title:

CELLULAR PLASTIC FROM DISPOSABLE

PRESSURIZED CONTAINERS

IPC:

C 08 G, C 08 L, C 08 J

The attached copies are a true and exact reproduction of the original documents of this patent application.

Munich, April 9, 2003

German Patent and Trademark Office

The President

by procuration

[signature]

HENKEL KGaA

TTP/Patents C/M/A

Dr. Mathes/Ge

February 21, 1994

#### PATENT APPLICATION

#### H 1215

### CELLULAR PLASTIC FROM DISPOSABLE PRESSURIZED CONTAINERS

The invention concerns a composition for producing cellular plastics from disposable pressurized containers as well as the cellular plastics themselves and their use.

Cellular plastics are materials with a cellular structure and consist, for example, of PU, PS, PE, or PVC. They are formed either by pressureless foam production (e.g., mechanically) or by sudden expansion of polymers or prepolymers that contain a (e.g., liquefied) gas. If the cellular plastic is produced at the site of its use, it is called an in-situ foam (DIN 18159). The composition to be foamed is usually held in disposable pressurized containers (aerosol cans). In-situ polyurethane foams are used chiefly in the construction industry for the sealing, insulation, and construction of, for example, joints, roof panels, windows, and doors.

The production of polyurethane foams from disposable pressurized containers is well known. In this process, a prepolymer that contains isocyanate groups is produced by reacting polyols with organic diisocyanates and/or polyisocyanates in the presence of a foam stabilizer and a catalyst and possibly plasticizers, flame retardants, and other ingredients. This reaction takes place in the presence of a liquefied propellant gas in a pressurized

container. After completion of the prepolymer formation, the foam can be discharged by metering it through a valve. The foam has a creamy consistency and cures by the action of environmental moisture, e.g., from the air, accompanied by an increase in volume (one-component foam). An activator may also be added from another pressurized container immediately before use. This causes faster, tack-free, thorough curing of the foam (two-component foam). The activator may be a short-chain diol, e.g., ethylene or propylene glycol, 1,4-butylene glycol, or glycerol.

A primary product of this kind manufactured for the production of one-component polyurethane foams is described in DD 298 647. To improve the fire behavior and to reduce the number of voids, the cited document proposes that the polyol of the prepolymer that contains isocyanate groups be a mixture of 8-40 wt.% of polyethylene glycol with an average molecular weight of 500 to 1,000 g/mole and 60-92 wt.% of a polyether alcohol that contains tertiary amino groups.

DE 40 25 843 describes a one-component mixture that is stable in storage for the production of polyurethane foam. This mixture contains a prepolymer with a dynamic viscosity of 200 to 4,000 mPa·s, measured at 20°C, and a content of NCO groups of 13-15 wt.%. Here too, the formation of the prepolymer takes place in the aerosol can. DE 39 11 784 describes a similar process for producing the prepolymer either directly in the aerosol can or in another pressurized container.

The above applications solved an environmental problem, namely, instead of using propellant gases such as chlorofluorocarbons, fluorocarbons, chlorofluorohydrocarbons, and fluorohydrocarbons, more environmentally compatible propellants gases ( $CO_2$  and  $NO_2$ ) were used.

Another critical point is the composition of the NCO prepolymer. Specifically, it is produced, practically without exception, directly in the

pressurized containers from mixtures of technical diphenylmethane-4,4'diisocyanate (MDI) with a functionality of 2.3 to 2.7 and polyols with an NCO:OH ratio of 3-10:1, and preferably 4-6:1, in the presence of a tertiary amine as catalyst. Due to the excess of MDI, a large amount of free, unreacted MDI is present, specifically, on the order of 7-15 wt.%, based on the total content of the pressurized container. Due to this content of monomeric MDI, the compositions must carry the label "low-toxicity, contains diphenylmethane-4,4'-diisocyanate" and St. Andrew's cross as a hazard symbol. If more highly volatile polyisocyanates were used instead of MDI to produce the prepolymer, the reaction mixtures would also contain large amounts of unreacted diisocyanate. In accordance with the Hazardous Substances Act, these products would then have to carry the label "poison" and the death'shead as a hazard symbol. Due to this increased toxicity, diisocyanates of this type were not used in insulation and structural foams from aerosol cans. Furthermore, the curing times of prepolymers produced from aliphatic or alicyclic diisocyanates are too long for use as one-component insulation and structural foams. Therefore, only MDI is actually used for this application.

The cellular plastics produced from the prepolymers pose no problems, since the free MDI reacts with water and in this way is permanently combined as a urea unit with the crosslinked polyurethane.

However, the disposal of the residual prepolymers in the disposable pressurized containers does present a problem. In accordance with applicable waste disposal regulations in Germany, they must be disposed of as special waste. The disposal costs continue to rise due to limited waste disposal space. Therefore, there is a need for insulation and structural foams whose residues or waste can be easily disposed of.

An obvious solution would be to produce insulation and structural foams from polymers other than PU, e.g., from polystyrene.

However, the solution proposed in accordance with the invention is that the composition with the isocyanate prepolymer as the reactive component, which remains in the pressurized container after use, has a content of diisocyanate monomers of less than 2.0, especially less than 1.0, and most especially less than 0.5 wt.% at the latest 24 hours after the foaming. Preferably, these values are reached after only 2 hours or even 0.5 hours. It is advantageous for the composition to have a content of diisocyanate monomers this low even before the foaming.

Preferably, this is accomplished by ensuring that the prepolymer already has a diisocyanate content this low before the foaming, e.g., because it was distilled. However, it can also be effective to polymerize the diisocyanate monomers by adding trimerization catalysts immediately before or after the foaming. In the case of one-component systems, it is also possible to add an OH compound, especially a monohydric alcohol, to the residual composition after the foaming.

The composition necessarily consists of at least one isocyanate prepolymer, at least one catalyst for the reaction of the isocyanate group with the OH group, at least one propellant [They have already used Treibgas to mean propellant gas. We thought they might be using Treibmittel to mean blowing agent rather than propellant, but polyurethane foams are self-blowing, i.e., the excess isocyanate reacts with environmental moisture to produce  $CO_2$ , which produces the cells in the plastic. On p. 10 we find out that the Treibmittel are all typical propellants, such as tetrafluoroethane, butane, etc. So apparently there is no blowing agent, only a propellant. This is consistent with the statement made above that the Treibmittel is a necessary component and the statement in Whittington's Dictionary of Plastics that blowing agents are not necessary components, although they are sometimes used.

-- Tr. Ed.] and at least one foam stabilizer. Other additives may also be

used, e.g., solvents, flame retardants, plasticizers, cell regulators, and antiaging agents.

An "isocyanate prepolymer" is understood to mean an oligomer with reactive NCO groups, which is involved as a preadduct in the synthesis of the polymer. The isocyanates are preferably aliphatic diisocyanates with 2-36, and especially 4-7, C atoms or alicyclic diisocyanates with 5-30, and especially 8-15, C atoms. However, aromatic diisocyanates with 8-20, and especially 8-11, carbon atoms may also be used. The diisocyanates should boil no higher than at  $180^{\circ}\text{C}$  at 10~mbars. Concrete examples of suitable diisocyanates include: hexamethylene diisocyanate (HDI), tetramethylene diisocyanate (TMDI), isophorone diisocyanate (IPDI), toluene-2,6-diisocyanate (2,6-TDI), toluene-2,4-diisocyanate (TDI), m-tetramethylxylene diisocyanates (m-TMXDI), p-tetramethylxylene diisocyanates (p-TMXDI), trimethylhexamethylene diisocyanate (TMDI), dimeryl [sic? "dimethyl"? -- Tr. Ed.] diisocyanate (DDI), p-phenylene diisocyanate (PPDI), 1,5-naphthalene diisocyanate (NDI), diphenylmethane-4,4'-diisocyanate (MDI), tolidine diisocyanate (TODI), bis(4isocyanatocyclohexyl)methane (H12-MDI), 3(4)-isocyanatomethyl-1-methylcyclohexyl isocyanate (IMCI), phenyl isocyanate, and ester isocyanates from isocyanatocarboxylic acid chlorides and silylated polyalcohols (see Mormann: Tetrahedron Letters, 28 (1987), 3087 ff. and Mormann: Makromol. Chem., Makrom. Symp. 25 (1989) 117 ff.).

Preferred diisocyanates are those whose NCO groups have different reactivities. They make it possible to produce monomer-poor prepolymers from polyols without distillation. Diisocyanates of this type are, for example, isophorone diisocyanate and toluene diisocyanate. Prepolymers from IPDI and TMP (trimethylolpropane) are preferred, provided they have been produced in monomer-poor form. A process for producing polyurethane prepolymers with a low residual monomer content is described in EP 0,150,444. In a first

reaction step, the diisocyanate is reacted with a polyfunctional alcohol using an OH:NCO ratio of between 4 and 0.55. After practically all fast NCO groups have reacted with a portion of the available OH groups, a more reactive diisocyanate, i.e., relative to the less reactive NCO groups of the isocyanate of reaction step I, is added in an equimolar amount of in slight excess relative to the free OH groups that are still present. If desired, catalysts may be added, or higher temperatures may be used. The content of EP 0,150,444 is explicitly incorporated in the present application.

The diisocyanates may also be replaced up to a molar fraction of 40%, and especially 20%, by mono- or tri-isocyanates. Concrete examples include: phenyl isocyanate [this is only one example! -- Tr. Ed.].

Isocyanate prepolymers can be produced from the diisocyanates without an additional reactive component by trimerizing the diisocyanates to form isocyanurates. As is well known, this reaction occurs in the presence of suitable trimerization catalysts (see, e.g., Kunststoff-Handbuch [Plastics Handbook], Vol. 7, Polyurethanes, p. 108). Mixtures of cyclotrimers produced from aliphatic and alicyclic diisocyanates are especially advantageous, especially mixed trimers produced from them.

However, the isocyanate prepolymers may also be produced by reaction of diisocyanates with polyols in the presence of suitable catalysts. Suitable catalysts are those which accelerate the reaction of the isocyanate group with the OH group but do not accelerate its trimerization. Concrete examples are dimorpholinodiethyl ether, bis(dimethylaminoethyl)ether, Dabco X-DM (Air Products Co.), and N-ethyl morpholine. However, other catalysts may also be considerd, if they do not trimerize the isocyanate groups during storage, e.g., N-substituted morpholines and their mixtures with propylene oxide adducts of triethanolamine, as well as the well-known metal catalysts, especially tin catalysts.

Polyols that may be used to produce the prepolymers include all customary polyesters and polyethers that contain hydroxyl groups.

Polyesters that may be used include esters of dicarboxylic acids, preferably aliphatic dicarboxylic acids with 4-8 carbon atoms in the alkylene group, which are reacted with polyhydric alcohols, preferably diols, such that these also must have free OH groups for the reaction. Examples of aliphatic dicarboxylic acids are pimelic acid, glutaric acid, azelaic acid, sebacic acid, and preferably succinic acid and adipic acid, and examples of aromatic dicarboxylic acids are phthalic acid and terephthalic acid. Dihydric and polyhydric alcohols that may be used include ethylene glycol, diethylene glycol, 1,2- or 1,3-propylene glycol, dipropylene glycol, glycerol, trimethylolpropane, as well as 1,4-butanediol and 1,6-hexanediol.

However, it is also possible to use polyester polyols of oleochemical origin, which have no free epoxy groups and have been produced by complete ring opening of epoxidized triglycerides of a fatty acid mixture that contains at least a partially olefinically unsaturated fatty acid with one or more alcohols with 1-12 C atoms and subsequent partial transesterification of the triglyceride derivatives to alkyl ester polyols with 1-12 C atoms in the alkyl group (see DE 36 26 223). Products produced by the well known method from one or more alkylene oxides with 2-4 carbon atoms in the alkylene group and a starter molecule that contains 2-4 active hydrogen atoms may be used as polyethers. Examples of suitable alkylene oxides are tetrahydrofuran, 1,3propylene oxide, 1,2- to 2,3-butylene oxide, and ethylene oxide. Possible starter molecules are: water, dicarboxylic acids, polyhydric alcohols, such as ethylene glycol, 1,2-propylene glycol, diethylene glycol, dipropylene glycol, glycerol, trimethylolpropane, pentaerythritol, sorbitol, and saccharose, as well as compounds that contain amino groups. Other polyols are: polycarbonate polyols and dimer [sic? "dimeric" -- Tr. Ed.] diols

(Henkel Co.).

The isocyanate polymers are produced from the diisocyanates and the polyols by well-known means. To produce monomer-poor isocyanate prepolymers, the volatile isocyanates, which are present in excess, are distilled off at temperatures of 100-160°C in a vacuum with the use of a thin-layer or flash evaporator. More concrete information on the production of monomer-poor isocyanate prepolymers by distillation is given, e.g., in DE 41 40 660, which describes the production of polyisocyanates that contain ether and urethane groups on the basis of polyhydroxy polyethers and toluene diisocyanate. These polyisocyanates have an NCO content of 11.8 to 14.4 wt.%, a mean NCO functionality of 3.1 to 4.0, and a content of free toluene diisocyanate of less than 0.1 wt.%. The content of the cited document is explicitly incorporated as part of the content of the present patent application to the extent that it refers to the production of the prepolymers. However, the use of the prepolymers specified there for the production of polyurethane coatings is not incorporated in the present patent application.

The production of certain polyadducts of TDI with residual monomer concentrations of at least 0.2% is also described in the patent documents DE 15 95 273 and US 4,128,825. Again, the production of these prepolymers is explicitly incorporated in the present application.

Of the two possibilities for producing monomer-poor isocyanate prepolymers, distillation is preferred to production with differently reactive diisocyanate groups. As a result, the prepolymers may then be produced outside the pressurized container rather than in the pressurized container, as has been customary until now.

Regardless of how they are produced, the monomer-poor isocyanate prepolymers have the following characteristics: They have an isocyanate monomer content of less than 3.0, preferably less than 1.0, and especially

less than 0.5 wt.%, based on the prepolymer. They have an NCO functionality of 2-5, and especially 2.5-4.2, and an NCO content of 8-30 wt.%, and especially 10-23 wt.%, based on the prepolymer, as well as a viscosity of 5-200, and especially 10-100, Pa·s at 25°C in accordance with DIN 53015. The prepolymers are preferably produced from aliphatic diisocyanates with 2-12, and preferably 4-8, C atoms and from alicyclic isocyanates with 5-30, and preferably 7-12, C atoms. However, it is also possible to use aromatic diisocyanates with 8-20 C atoms. The boiling point of the diisocyanates should be a maximum of 180°C at 10 mbars, and preferably a maximum of 160°C at 10 mbars.

As is customary, these isocyanate prepolymers are mixed in the pressurized containers with catalysts for the reaction of the isocyanate groups with OH groups, with propellants, and with foam stabilizers. In addition, customary additives may be used, such as solvents, flame retardants, plasticizers, cell regulators, and antiaging agents. A solution or emulsion is formed.

Bis(dimethylaminoethyl)ether is preferably used as the catalyst. It is intended to catalyze only the reaction of the NCO group with OH groups, but not its trimerization during storage.

Preferred propellants are 1,1,1,2-tetrafluoroethane, 1,1-difluoroethane, and dimethyl ether. However, n-propane, n-butane, and isobutane may also be used.

Siloxane-oxyalkylene copolymers, e.g., Tegostab B 8404 (Goldschmidt Co.), are preferably used as foam stabilizers.

The contents of the pressurized containers preferably have the following quantitative composition:

- -- 50-90, and preferably 60-85 wt.%, of isocyanate prepolymers,
- -- 0.1-5.0, and preferably 0.5-20 [sic? "0.5-2.0" -- Tr. Ed.], wt.% of

catalysts,

- -- 5-35, and preferably 10-25, wt.% of propellants, and
- -- 0.1-5.0, and preferably 0.5-3.0, wt.% of foam stabilizers.

Of the various optional additives, the flame retardant may be added in an amount of 5-50, and preferably 10-30, wt.%. The other optional additives may be added in an amount of 0.1-3.0, and especially 0.2-1.5, wt.%. The above specifications in wt.% are based on the total composition.

Besides these compositions with a very low content of diisocyanates, standard compositions with the usual amounts of isocyanates may also be used, if at least one trimerization catalyst, which can also accelerate the moisture curing, is added to them immediately before their use (foaming). Concrete examples of these kinds of catalysts are dibutyltin dilaurate, potassium acetate, potassium 2-ethylhexoate, N,N-dimethylcyclohexylamine, and tris-2,4,6-(dimethylaminomethyl)phenol. This ensures not only that the curing of the foam is accelerated, but also that the NCO prepolymer and monomer in the container are converted within one day to the polymeric isocyanurate. After this amount of time, the residual reaction mixture contains almost no more monomeric diisocyanate. After this reaction has occurred, the compressed gas containers may be disposed of and recycled as ordinary containers, because they are no longer to be regarded as hazardous material after 24 hours at the latest.

The trimerization catalyst must be stored separately from the rest of the composition and be added and mixed immediately before the foaming. Suitable aerosol containers for this are well known (see, e.g., EP 0,024,659 or DE 36 10 345).

To be certain that the amine catalyst and the composition are sufficiently mixed, it is advisable to mix the amine catalyst with a dye and/or pigment. If the mixing was incomplete or there was no mixing, then the

foam will not be uniformly colored or will not be colored at all.

In another modification of the invention, the normal catalyst needed for the moisture curing is mixed in during the filling of the cans. After the can is empty, another catalyst is added to the residual amount of isocyanate prepolymer still present in the pressurized container, so that it trimerizes within a short amount of time and is thus converted to a "nonhazardous material". Specifically, this leads to the formation of brittle, high-molecular-weight polyisocyanurates. If low-molecular-weight monohydric alcohols, e.g., ethanol and propanol, are added in excess, plasticizer-like urethanes are formed. If diols with a molecular weight of < 400 are added in excess, oligomeric polyurethanes with OH terminal groups are formed. Both are likewise nonhazardous substances.

Compressed gas containers suitable for this are also well known and are used, e.g., for two-component polyurethane foams. The trimerization catalyst, the monohydric alcohol, or the diol is preferably released automatically after the normal processing of the foam. Of course, special containers are necessary for this, as described, for example, in EP 446,973 and EP 349,053. It might be mentioned, for example, that the pressurized container can contain another, small pressurized container, which contains the catalyst and automatically empties when the ambient pressure in the large pressurized container falls below 2.5 bars due to the removal of the foam.

The compositions of the invention make it possible to produce a one-component plastic foam that cures in the usual way with environmental moisture. However, a two-component plastic foam is also immediately possible, if a polyol is added to the composition in preferably equivalent amounts or slightly deficient amounts. These polyols are substances ordinarily used with 2-6 C atoms, and with 2 or 3, preferably primary, OH groups.

The plastic foam produced in this way is suitable especially as an

insulating foam, structural foam, or sealing foam in the construction industry, especially for in-situ use.

The invention is illustrated by the following examples:

#### EXAMPLES

#### Example 1

From 191 g of a commercial cyclotrimer of 1,6-diisocyanatohexane, which has an NCO content of 22.6 wt.% (= 1 mole of NCO) and is sold under the trade names Tolonate HDT (Rhône-Poulenc) and Desmodur N 3300 (Bayer), a high-viscosity adduct with a viscosity of ca. 10 Pa·s at 25°C (DIN 53015) was produced by addition of 0.05 mole of ethylene glycol (= 0.1 mole of OH). 2.0 g of a commercial silicone surfactant with the trade name Tegostab B 8400 (Goldschmidt Co.) and 2.0 g of a catalyst with the trade name Texacat CD (chemical name: bis(N, N'-dimethylaminoethyl)ether) were added to 96 g of this NCO prepolymer, and a disposable pressurized container was filled with this mixture. 25.0 g of dimethyl ether and 10.0 g of tetrafluoroethane (fluorohydrocarbon 134a) were added to the pressurized container, which was then shaken until the NCO prepolymer was dissolved in the propellant gas mixture. The concentration of monomeric HDI was < 0.1 wt.%, based on the total composition.

The dissolved product was then removed from the pressurized container and introduced into a joint with dimensions of  $3.0 \times 5.0 \times 50.0$  cm at a temperature of 25°C and a relative humidity of 50% and allowed to cure. The resulting foam was characterized by the following data:

| tack-free time of the surface | 10 minutes |
|-------------------------------|------------|
| time for complete curing      | 2 hours    |

| ca. 24 g/L |
|------------|
| elastic    |
|            |

### Examples 2 to 9

In the following examples, in addition to the HDI cyclotrimer, a commercially available IPDI cyclotrimer was used, which is sold by Hüls under the trade name Vestanate T 1890/100 (NCO content: 17.0 to 17.5 wt.%; melting range: 100-115°C; monomer concentration: < 0.7 wt%).

|    | 2           | 3  | 4   | 5  |
|----|-------------|--|---|--|
| g  | 90.0        | 80.0   | 64.0  | 48.0   |
| g  | 10.0        | 20.0   | 32.0  | 48.0   |
| g  | 2.0         | 2.0  | 2.0   | 2.0  |
| g  | 2.0         | 2.0  | 2.0   | 2.0  |
| g  | 6.5         | 6.5  | 15.0  | 15.0   |
|    | 19.5        | 19.5   | 10.0  | 10.0   |
| g  | 130.0       | 130.0  | 125.0   | 125.0  |
|    | 9           | 9  | 10  | 10   |
|    | 2           | 2  | 2   | 2  |
|    | 22          | 24   | 24  | 23   |
|    | w/hh        | hh   | hh  | h  |
| 9  | < 1         | < 1  | 20  | < 5  |
| at | > 4         | > 4  | > 4   | > 4  |
|    | a<br>a<br>a | g 90.0 g 10.0 g 2.0 g 2.0 g 6.5 g 19.5 g 130.0 g 22 w/hh % < 1 | g 90.0 80.0 g 10.0 20.0 g 2.0 2.0 g 2.0 2.0 g 6.5 6.5 g 19.5 19.5 g 130.0 130.0 9 9 2 2 22 24 w/hh hh % < 1 < 1 | g       90.0       80.0       64.0         g       10.0       20.0       32.0         g       2.0       2.0       2.0         g       6.5       6.5       15.0         g       19.5       19.5       10.0         g       130.0       130.0       125.0         g       9       9       10         2       2       2         22       24       24         w/hh       hh       hh         %       < 1 |

| example number | 6 | 7 | 8 | 9 |
|----------------|---|---|---|---|
|                |   |   |   |   |

|                               |      | - 00 0 [ | 90.0  | 90.0  | 90.0  |
|-------------------------------|------|----------|-------|-------|-------|
| HDI trimer                    | g    | 90.0     | 90.0  | 50.0  |       |
| IPDI trimer                   | g    | 10.0     | 10.0  | 10.0  | 10.0  |
| silicone surfactant           | g    | 2.0      | 2.0   | 2.0   | 2.0   |
| Texacat ZF-20                 | g    | 2.0      | 2.0   | 2.0   | 2.0   |
| Baysilon oil M 1001)          | g    |          | 0.02  |       |       |
| dimethyl ether                | g    | 6.5      | 6.5   |       |       |
| 1,1,1,2-<br>tetrafluoroethane |      | 19.5     | 19.5  |       | 21.0  |
| 1,1-difluoroethane            | g    |          |       | 26.0  | 5.0   |
| total                         | g    | 130.0    | 130.0 | 130.0 | 130.0 |
| tack-free time, min2          | !)   | 9        | 9     | 9     | 9     |
| curing time, h <sup>3)</sup>  |      | 2        | 2     | 2     | 2     |
| gross density, $g/L^{4)}$     |      | 23       | 25    | 22    | 25    |
| foam hardness <sup>5)</sup>   |      | hh       | hh    | hh    | hh    |
| dimensional change,           | g 6) | > 30     | 9     | 15    | 8     |

## Notes on the Preceding Tables

- $^{1)}$  Baysilon oil M 100 is a polydimethylsiloxane with terminal trimethylsiloxy groups and a viscosity of 140  $\rm mm^2 s^{-1}$  at 20°C produced by the Bayer Co.
- $^{2)}$  The tack-free time is the time from the beginning of foaming until the foam is no longer tacky.
- $^{3)}$  The curing time is the time from the beginning of foaming until the foam can be cut with a knife without residual foam sticking to the knife.
- $^{4)}$  The gross density was determined by the SKZ method (SKZ = Southern German Plastics Center).
- $^{5)}$  The foam hardness was determined by compression tests in accordance with DIN 53421.

The evaluation was based on the compressive stress at 10% compressive strain,

where h (= hard) means that this occurs at > 10 N/cm<sup>2</sup>,

hh (= semihard) means that this occurs at 1-10  $\rm N/cm^2$ , and w (= soft) means that this occurs at < 1  $\rm N/cm^2$ .

<sup>6)</sup> The dimensional change was determined by the SKZ method (= test specifications for polyurethane structural foam, published by the Southern German Plastics Center (SKZ) in July 1982).

 $^{7)}$  The storage stability was determined as follows:

The viscosity of the samples was determined with a rotational viscometer (Brookfield RVT, spindle 7, 50 rpm, 25°C). The prepolymer was then stored at 50°C and at intervals of 1 week was briefly cooled to 25°C to remeasure the viscosity. The time until tripling of the initial viscosity is defined as the storage stability. "w" means weeks.

8) The content of diisocyanate monomers in the prepolymer and in the composition is determined by HPLC (high-pressure liquid chromatography). [We can't find "8)" in the text or tables, but presumably they meant to place it at the ends of the paragraphs on Example 1 and Examples 2-9, where they mention the concentration of monomer -- Tr. Ed.]

#### CLAIMS

- 1. Composition for producing cellular plastics from disposable pressurized containers, characterized by the fact that the residual composition remaining in the pressurized container has a concentration of disocyanate monomers of less than 2.0 wt.%, based on the residual content of the emptied container, at the latest one day after use.
- 2. Composition in accordance with Claim 1, characterized by the fact that its content of diisocyanate monomers is less than 1.0 or 0.5 wt.%, based on the total content of the container, and preferably before it is used.
- 3. Composition in accordance with Claim 1 or Claim 2, characterized by the fact that it contains the following components before it is used:
- (A) at least one isocyanate prepolymer as the reactive component with a concentration of diisocyanate monomers of less than 3.0 wt.%, based on the prepolymer, with an NCO functionality of 2 to 5, with an NCO content of 8 to 30 wt.%, based on the prepolymer, and with a viscosity of 5 to 200 Pa·s at 25°C in accordance with DIN 53015, such that the prepolymer was produced from aliphatic diisocyanates with 2 to 36 C atoms, alicyclic diisocyanates with 5 to 30 C atoms, and/or aromatic diisocyanates with 8 to 20 C atoms, in each case with a boiling point of a maximum of 180°C at 10 mbars,
- (B) at least one catalyst for the reaction of the isocyanate group with OH groups,
  - (C) at least one propellant,
  - (D) at least one foam stabilizer, and
- (E) possibly additives, such as solvents, flame retardants, and plasticizers.
- 4. Composition in accordance with Claim 3, characterized by the fact that the reactive component is a cyclotrimer of a diisocyanate (isocyanurate),

especially mixtures of HDI and IPDI and their mixed trimers.

- 5. Composition in accordance with Claim 3, characterized by the fact that the reactive component is a prepolymer produced from diisocyanates and/or isocyanurates with NCO groups and polyols.
- 6. Composition in accordance with one or more of Claims 1 to 5, characterized by the fact that the prepolymer was produced from diisocyanates with different reactivities of the NCO groups.
- 7. Composition in accordance with one or more of Claims 1 to 6, characterized by the following quantitative specifications:
  - -- 50 to 90 wt.% of the prepolymer,
  - -- 0.1 to 5.0 wt.% of the catalyst,
  - -- 5 to 35 wt.% of the propellant,
  - -- 0.1 to 5.0 wt.% of the foam stabilizer, and
  - -- 0 to 51.5 wt.% of additives.
- 8. One-component plastic foam, which can be produced from the composition in accordance with one or more of Claims 1 to 7 and moisture.
- 9. Two-component plastic foam, which can be produced from the composition in accordance with one or more of Claims 1 to 7 as the first component and a polyol as the second component.
- 10. Plastic foam in accordance with Claim 8 or Claim 9, characterized by its use as an insulation foam or structural foam, especially in situ.

#### ABSTRACT

## CELLULAR PLASTIC FROM DISPOSABLE PRESSURIZED CONTAINERS

A composition for producing cellular plastics from disposable pressurized containers is described, which is characterized by the fact that the residual composition remaining in the pressurized container has a concentration of disocyanate monomers of less than 2.0 wt.%, based on the residual content of the emptied container, at the latest one day after use (foaming). Due to this concentration reduction by a factor of > 5, the residual composition is far less harmful to the environment than it would otherwise be.